Structure of Hexakis(2-fluorophenylamino)disiloxane

By M. Jansen and I. Mokros

Institut für Anorganische Chemie, Universität Bonn, Gerhard-Domagk-Strasse 1, D-W-5300 Bonn 1, Germany

Abstract. Hexakis(2-fluorophenylamino)disiloxane, $C_{36}H_{30}F_6N_6OSi_2$, $M_r = 732.83$, triclinic, $P\bar{1}$, $a = 9.160$ (1), $b = 9.921$ (2), $c = 10.372$ (4) Å, $\alpha = 100.57$ (2), $\beta = 112.01$ (2), $\gamma = 92.17$ (1)°, $V = 853.1$ Å^3, $Z = 1$, $D_x = 1.426$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.1703$ mm^{-1}, $F(000) = 378$, $T = 295$ K, $R = 0.061$, $wR = 0.048$ for 2104 observed reflections ($F_o/\sigma_F > 4$). The structure is comprised of isolated molecules which lie across a crystallographic centre of inversion. The Si—O—Si bridge is linear with shortened Si—O bond lengths (1.61 Å) and relatively low thermal motion of oxygen.

Experimental. Colourless crystals obtained from aminolysis of silicon tetrachloride (Aldrich, 99%) with 2-fluoroaniline (Aldrich, 99%) containing 2% water and triethylamine (Fluka, 99.5%) in toluene. Crystal dimensions 0.3 × 0.4 × 0.2 mm. Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation, cell dimensions determined from 2θ angles for 24 reflections in the range $10.18 < 2\theta < 24.70°$. Intensities measured up to $2\theta = 54°$ with h 12 to -12, k 13 to -13, l 13 to -13, scan type $\omega/2\theta$, two standard reflections monitored every 100 measurements showed no significant change. 8448 total reflections measured, 3653 unique ($R_{int} = 0.013$), with 2104 intensities observed ($F_o/\sigma_F > 4.00$). Neutral-atom scattering factors were taken from Cromer & Mann (1968). Source of anomalous-dispersion coefficients was Cromer & Liberman (1970). No absorption and no extinction correction. Data were corrected for Lorentz and polarization effects. Calculations were performed on a MicroVAX 3100 computer. The structure was solved by direct methods with *SHELX*76 and *SHELXS*86 (Sheldrick, 1976, 1986). Full-matrix least-squares refinement on F using a weighting scheme $\{w = 3.0441[\sigma^2(F) + 0.000156F^2]^{-1}\}$. 234 parameters refined, S not calculated. Final $R = 0.061$, $wR = 0.048$. No significant shift in the final refinement (max. $\Delta/\sigma = 0.009$). The unassigned electron density peaks were max. $\Delta\rho = 0.626$, min. $\Delta\rho = -0.384$ e Å^{-3}. Anisotropic thermal parameters were used for all non-H atoms. H atoms, located from difference Fourier maps, were placed in idealized

Table 1. *Final atomic coordinates and equivalent isotropic temperature parameters* (Å^2) *with e.s.d.'s in parentheses*

U_{eq} is defined as the geometric mean of the diagonal components of the diagonalized matrix of U_{ij}.

	x	y	z	U_{eq}
Si	0.1230 (1)	0.4172 (1)	0.9488 (1)	0.039 (1)
O	0.0000	0.5000	1.0000	0.041 (1)
F(1)	0.3642 (3)	0.1845 (2)	1.2691 (2)	0.082 (1)
F(2)	−0.1243 (3)	0.1423 (2)	0.5315 (2)	0.081 (1)
F(3)	0.4602 (2)	0.7283 (2)	0.9293 (2)	0.068 (1)
N(1)	0.1954 (3)	0.2993 (3)	1.0501 (3)	0.043 (1)
N(2)	0.0353 (3)	0.3309 (3)	0.7748 (3)	0.048 (1)
N(3)	0.2795 (3)	0.5284 (3)	0.9660 (3)	0.044 (1)
C(1)	0.1173 (4)	0.2134 (3)	1.1030 (4)	0.039 (2)
C(2)	0.2035 (4)	0.1568 (4)	1.2173 (4)	0.049 (2)
C(3)	0.1372 (5)	0.0781 (4)	1.2801 (4)	0.060 (2)
C(4)	−0.0261 (5)	0.0526 (4)	1.2272 (4)	0.060 (2)
C(5)	−0.1172 (5)	0.1032 (4)	1.1115 (4)	0.055 (2)
C(6)	−0.0460 (4)	0.1815 (3)	1.0486 (4)	0.043 (1)
C(7)	−0.0651 (4)	0.3740 (4)	0.6538 (4)	0.041 (1)
C(8)	−0.1494 (4)	0.2769 (4)	0.5296 (4)	0.049 (2)
C(9)	−0.2552 (5)	0.3085 (4)	0.4088 (4)	0.059 (2)
C(10)	−0.2802 (5)	0.4435 (5)	0.4084 (4)	0.065 (2)
C(11)	−0.1963 (5)	0.5443 (4)	0.5276 (4)	0.064 (2)
C(12)	−0.0888 (4)	0.5094 (4)	0.6492 (4)	0.051 (2)
C(13)	0.3710 (4)	0.6336 (4)	1.0812 (4)	0.041 (1)
C(14)	0.4627 (4)	0.7365 (4)	1.0632 (4)	0.051 (2)
C(15)	0.5533 (5)	0.8455 (5)	1.1692 (5)	0.075 (2)
C(16)	0.5539 (5)	0.8531 (5)	1.3035 (5)	0.084 (2)
C(17)	0.4660 (5)	0.7531 (5)	1.3277 (4)	0.067 (2)
C(18)	0.3763 (4)	0.6446 (4)	1.2189 (4)	0.051 (2)

positions with fixed common isotropic temperature coefficients. The final atomic coordinates and equivalent isotropic temperature parameters are given in Table 1.* Bond distances and angles are listed in Table 2. A perspective view of the molecule is presented in Fig. 1.

Related literature. The structure of the title compound reported here is comparable with crystal structures of [Ph(CH$_2$)$_3$$M$]$_2$O ($M$ = Si, Ge, Sn) investigated by Glidewell & Liles (1978, 1979, 1981). The Si—O bond distances of 1.608 (1) Å are compa-

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55390 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BX0582]

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Si(1)—O(1)	1.608 (1)	C(4)—C(5)	1.375 (6)
Si(1)—N(1)	1.703 (3)	C(5)—C(6)	1.386 (6)
Si(1)—N(2)	1.711 (3)	C(7)—C(8)	1.388 (4)
Si(1)—N(3)	1.709 (3)	C(7)—C(12)	1.376 (5)
F(1)—C(2)	1.361 (4)	C(8)—C(9)	1.365 (5)
F(2)—C(8)	1.366 (4)	C(9)—C(10)	1.368 (6)
F(3)—C(14)	1.368 (5)	C(10)—C(11)	1.377 (5)
N(1)—C(1)	1.400 (5)	C(11)—C(12)	1.391 (5)
N(2)—C(7)	1.401 (4)	C(13)—C(14)	1.380 (6)
N(3)—C(13)	1.394 (4)	C(13)—C(18)	1.395 (6)
C(1)—C(2)	1.386 (5)	C(14)—C(15)	1.371 (5)
C(1)—C(6)	1.386 (5)	C(15)—C(16)	1.380 (8)
C(2)—C(3)	1.360 (7)	C(16)—C(17)	1.371 (7)
C(3)—C(4)	1.382 (6)	C(17)—C(18)	1.378 (5)

N(2)—Si(1)—N(3)	108.3 (2)	N(2)—C(7)—C(12)	124.0 (3)
N(1)—Si(1)—N(3)	108.0 (2)	N(2)—C(7)—C(8)	119.8 (3)
N(1)—Si(1)—N(2)	108.0 (2)	C(8)—C(7)—C(12)	116.2 (3)
O(1)—Si(1)—N(3)	110.3 (1)	F(3)—C(8)—C(7)	116.8 (3)
O(1)—Si(1)—N(2)	111.6 (2)	C(7)—C(8)—C(9)	123.9 (4)
O(1)—Si(1)—N(1)	110.4 (1)	F(3)—C(8)—C(9)	119.2 (3)
Si(1)—N(1)—C(1)	129.4 (3)	C(8)—C(9)—C(10)	118.7 (4)
Si(1)—N(2)—C(7)	130.8 (3)	C(9)—C(10)—C(11)	119.7 (4)
Si(1)—N(3)—C(13)	128.9 (3)	C(10)—C(11)—C(12)	120.3 (4)
H(3)—N(3)—C(13)	115.5 (3)	C(7)—C(12)—C(11)	121.0 (4)
N(1)—C(1)—C(6)	123.6 (3)	N(3)—C(13)—C(18)	124.0 (3)
N(1)—C(1)—C(2)	120.1 (4)	N(3)—C(13)—C(14)	120.4 (3)
C(2)—C(1)—C(6)	116.3 (3)	C(14)—C(13)—C(18)	115.6 (4)
F(2)—C(2)—C(1)	116.8 (4)	F(1)—C(14)—C(13)	116.9 (3)
C(1)—C(2)—C(3)	124.1 (4)	C(13)—C(14)—C(15)	124.5 (4)
F(2)—C(2)—C(3)	119.2 (4)	F(1)—C(14)—C(15)	118.6 (4)
C(2)—C(3)—C(4)	118.3 (4)	C(14)—C(15)—C(16)	118.0 (4)
C(3)—C(4)—C(5)	119.9 (4)	C(15)—C(16)—C(17)	119.9 (5)
C(4)—C(5)—C(6)	120.4 (4)	C(16)—C(17)—C(18)	120.7 (4)
C(1)—C(6)—C(5)	120.9 (4)	C(13)—C(18)—C(17)	121.3 (4)

rable with those in the benzyl analogue [1.613 (4) Å] (Glidewell & Liles, 1981). The bond angles about the Si atoms (Table 2) are all close to tetrahedral, whereas the angles Si—N—C show values of about 130°, which are typical for that combination of atoms (Wannagat, 1964).
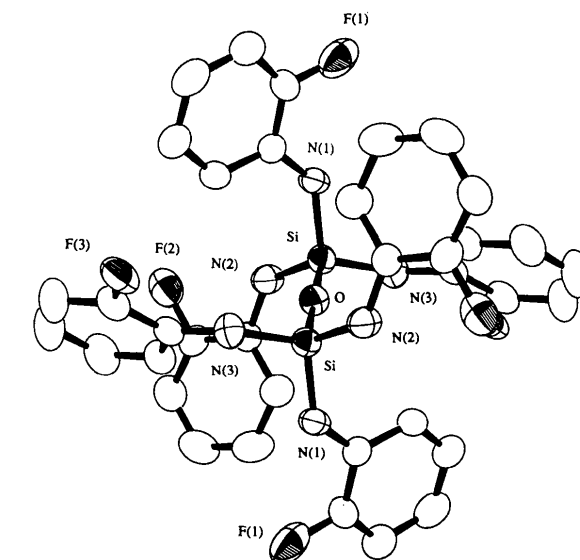

Fig. 1. Plot of the title compound showing the atomic numbering scheme with thermal ellipsoids drawn at the 50% level.

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Structure of 3,5-Dimethyl-2,2,4,4,6,6-hexakis(methylamino)-1-oxa-3,5-diaza-2,4,6-trisilacyclohexane

BY MARTIN JANSEN AND JOSUA LÖFFELHOLZ

Institut für Anorganische Chemie der Universität Bonn, Gerhard-Domagk-Strasse 1, D-5300 Bonn 1, Germany

Abstract. $C_8H_{30}N_8OSi_3$, $M_r = 338.6$, triclinic, $P\bar{1}$, $a = 8.984$ (6), $b = 14.899$ (20), $c = 16.561$ (22) Å, $\alpha = 113.00$ (7), $\beta = 99.63$ (7), $\gamma = 104.39$ (6)°, $V = 1888$ (4) Å^3, $Z = 4$, $D_x = 1.1914$ g cm^{-3}, λ(Mo $K\alpha$) $= 0.71069$ Å, $\mu = 2.53$ cm^{-1}, $F(000) = 736$, room temperature, $R_{int} = 0.01$, final $R = 0.060$, $wR = 0.062$ for 4928 observed reflections and 364 variables, $F(hkl) > 4\sigma(F)$. There are two independent molecules in the asymmetric unit. For each, the six-membered Si_3N_2O ring is planar. The C atoms of the methyl